D10

AutoPore IV 9500

Operator's Manual

SHIMADZU

micromeritics®

AutoPore IV 9500

Operator's Manual

Part No. 950-42801-01

Pop-up Menus 7-17
For Tabular Reports 7-17
For Graphs 7-18
Zoom Feature 7-21
Axis Cross Hair 7-21
Sample Reports 7-22
Graphs 7-22
Cumulative Intrusion vs. Pressure 7-23
Log Differential Intrusion vs. Pore Size 7-24
Tabular Reports 7-25
Cavity to Throat Size Ratio 7-26
Material Compressibility 7-27
Fractal Dimension 7-28
Summary 7-29

8. TROUBLESHOOTING AND MAINTENANCE

Troubleshooting 8-1
Preventive Maintenance 8-7
Maintaining Mercury Level 8-8
Draining Spilled Mercury Dish 8-9
Maintaining High Pressure Fluid Level 8-10
Maintaining Vacuum Pump Fluid Level 8-11
Greasing Low Pressure Ports 8-11
Replacing Chamber Plug Seals 8-12
Changing High Pressure Fluid and Cleaning the High Pressure Chambers 8-13
Maintaining Hydraulic Pump Fluid Level 8-13
Changing Vacuum Pump Fluid 8-14
Replacing Vacuum Pump Exhaust Filter 8-14
Checking the Valves for Leaks 8-14
Cleaning Valves 8-15
Removing Moisture from the System 8-16
Replacing the Banana Plug 8-17
High Pressure Chamber 8-17
Low Pressure Capacitance Detector 8-18

9. ORDERING INFORMATION

Appendix A: Forms
Appendix B: Theory
Appendix C: Proper Handling of Mercury
Appendix D: Data Reduction
Appendix E: Exported Data Format
Appendix F: Use of the Maximum Intrusion volume Option
Appendix G: Blank and Sample Compression Correction for Mercury Porosimetry

APPENDIX D

DATA REDUCTION

DATA REDUCTION

Data for presentation in tabular and plot form is calculated in the following manner:

P_i = head-corrected pressure as stored
V_{ri} = intrusion volume as stored
θ = user-entered contact angle
γ = user-entered surface tension
W_s = user-entered sample weight
W_p = user-entered weight for penetrometer
W_{psm} = user-entered weight for penetrometer + sample + mercury
V_p = user-entered volume for penetrometer
V_c = user-entered volume for capillary (stem)
Y_m = user-entered density for mercury

$$\text{WASHCON=Washburn constant} = \frac{10^4\ \mu m/cm}{68947.6\ \text{dynes/cm}^2\text{–psia}} = 0.145038$$

For all calculations requiring interpolation between collected data points, an Akima* method semi-spline is used.

Diameter for the i^{th} point is:

$$D_i = \frac{\text{WASHCON}\ \gamma(-4\cos\theta)}{P_i}$$

Radius for the i^{th} point is:

$$R_i = \frac{D_i}{2}$$

Cumulative specific intrusion volume for the i^{th} point is:

$$I_i = \frac{V_i}{W_s}$$

Mean diameter for the i^{th} point is:

$$Dm_i = \frac{D_i + D_{i-1}}{2}$$

* "A New Method of Interpolation and Smooth Curve Fitting Based on Local Procedures," *Journal of the Association of Computing Machinery*, 17(4) 1970, 589-602.

Incremental specific intrusion volume for the i^{th} point is:

$$Ii_i = I_i - I_{i-1}$$

Incremental specific pore area for the i^{th} point is:

$$Ai_i = \frac{4 \times Ii_i}{Dm_i}$$

Cumulative specific pore area for the i^{th} point is:

$$A_i = Ai_i + Ai_{i-1} + \ldots + Ai_1$$

If more than 8 data points are available, differential and log differential specific intrusion volume are calculated as follows.

Differential and log differential data are the 1st derivative of the cumulative specific intrusion volume (all) data as a function of calculated log diameter, normalized by the diameter or log diameter interval. This derivation is comprised of four transformations.

1. Interpolation of cumulative specific intrusion volume vs. log diameter is made to get cumulative specific intrusion volume corresponding to evenly spaced log diameters.

2. The uniform cumulative specific intrusion volume data are then subjected to a 1st derivative calculation, using a 9-point smoothing method. This gives the desired differential data in terms of uniform intervals of collected data.

3. Log differential data are normalized by dividing by the log diameter interval between points. Since the points are evenly log spaced, this interval is the same for all points. Differential data are normalized by dividing by the diameter interval between points. Since the points are evenly log spaced, this interval is larger for larger diameters.

4. Interpolation of the differential or log differential data vs. log diameter is made to get data corresponding to collected data points.

If 8 or fewer data points are available, differential and log differential specific intrusion volume are calculated as follows.

Differential specific intrusion volume by diameter for the i^{th} point is:

$$Id_i = \frac{-Ii_i}{D_i - D_{i-1}}$$

Log differential specific intrusion volume by diameter is:

$$Ild_i = \frac{-Ii_i}{\log D_i - \log D_{i-1}}$$

Differential specific intrusion volume by radius for the i^{th} point is:

$$Ir_i = \frac{-Ii_i}{R_i - R_{i-1}}$$

Log differential specific intrusion volume by radius is:

$$Ilr_i = \frac{-Ii_i}{\log R_i - \log R_{i-1}}$$

Total intrusion volume is:

$$V_{tot} = V_j$$

where the j^{th} point is the first such that:

$$P_{j+1} \le P_j - 10 \quad \text{and} \quad P_{j+1} \le P_j \times 0.995$$

Total specific intrusion volume is:

$$I_{tot} = \frac{V_{tot}}{W_s}$$

Percent of total specific intrusion volume for the i^{th} point is:

$$Ip_i = \frac{100 \times I_i}{I_{tot}}$$

Total specific pore area is:

$$A_{tot} = A_j$$

for point j as defined above.

Median diameter by volume is:

$$D_{mv} = D_k$$

where

$$I_k = \frac{I_{tot}}{2}$$

and P_k is interpolated from I_k and the collected data, and D_k is calculated from P_k.

Median diameter by area is:

$$D_{ma} = D_k$$

where

$$A_k = \frac{A_{tot}}{2}$$

and P_k is interpolated from A_k and the collected data, and D_k is calculated from P_k.

Average diameter is:

$$D_{av} = \frac{4 \times I_{tot}}{A_{tot}}$$

Blank Correction by Formula:

For equilibration time 6 seconds: $X = \log\left(\frac{T}{6}\right)$

SHIMADZU CORPORATION

SHIMADZU CORPORATION, International Marketing Division
3, Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan
Phone 81(3)3219-5641 Fax.81(3)3219-5710

Cable Add: SHIMADZU TOKYO

SHIMADZU SCIENTIFIC INSTRUMENTS, INC.
7102 Riverwood Drive, Columbia, Maryland 21046, U.S.A
Phone: 1(410)381-1227 Fax. 1(410)381-1222 Toll Free: 1(800)477-1227

SHIMADZU EUROPA GmbH
Albert-Hahn-Strasse 6-10, D-47269 Duisburg, F.R. Germany
Phone: 49(203)7687-0 Fax. 49(203)766625

SHIMADZU (ASIA PACIFIC) PTE LTD.
16 Science Park Drive #01-01 Singapore Science Park, Singapore 118227, Republic of Singapore
Phone: 65-6778 6280 Fax. 65-6779 2935

SHIMADZU SCIENTIFIC INSTRUMENTS (OCEANIA) PTY. LTD.
Units F, 10-16 South Street, Rydalmere N.S.W. 2116, Australia
Phone: 61(2)9684-4200 Fax. [illegible]

SHIMADZU DO BRASIL COMERCIO LTDA.
Avenida Marcaes de Sao Vicente,1771,Barra Funda CEP:01139-003-Sao Paulo-SP, Brasil
Phone: (55)11-3611-1688 Fax. (55)11-3611-2209

SHIMADZU (HONG KONG)LIMITED
Suite 1028 Ocean Center, Harbour City, Tsim,Sha Tsui,Kowloon HONG KONG
Phone: (852)2375-4979 Fax. (852)2199-7438

SHIMADZU INTERNATIONAL TRADING (SHANGHAI) Co.,LTD. PUXI BRANCH
Block F,No.570 West Huaihai Road,Shanghai,200052 P.R. of China
Phone: 86(21)2201-3188 Fax. 86(21)2201-3666

Overseas Offices
Istanbul, Moscow

URL http://www.shimadzu.com

SHIMADZU